doi:10.1017/S1431927617000952
Microsc. Microanal. 23 (Suppl 1), 2017

Stable and Flexible Side-Entry Stage for Nion STEMs

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Aberration-corrected scanning transmission electron microscopes (STEMs) can resolve single atoms, probe them spectroscopically, and produce elemental and chemical maps with atomic resolution in 2D and 3D. When equipped with a monochromator and operating in an aloof mode, STEMs can record vibrational electron energy loss spectra (vib-EELS) to create nm-scale vibrational maps while avoiding radiation damage. These techniques require excellent short-term (vibration) and long-term (drift) stability of the microscope's sample stage.

Typical side-entry stages are mounted on the side of the microscope in such a way that the sample is connected to the objective lens (OL) polepiece through a ~30 cm long mechanical path. This means that if the temperature of the microscope (or of the sample rod) changes by just 0.1° C then the sample can drift by hundreds of nm. This results in image drift rates of the order of nm per minute being common with side-entry stages in all but the most stable environments.

In order to minimize these kinds of problems, Nion's cartridge-based sample stage was designed to have an OL polepiece-to-sample mechanical path of 10 cm and a thermal expansion center that coincides with the microscope's optic axis [1]. The detachable sample cartridges have no direct link to the microscope's outside. These two innovations have resulted in drift rates smaller than 1 nm per hour and sample vibration amplitudes <0.1 Å r.m.s. [2]. Unfortunately, compared to a side-entry sample rod that supports the sample at one end and exits outside the microscope vacuum at the other end, a detachable cartridge in not as well suited for bringing in "services" to the sample such as cooling, heating, electrical, gases, liquids, straining, tilting, and nano-indenting.

To combine the stability and freedom from drift of our centro-symmetric design with the flexibility of a side-entry sample rod, Nion has developed a new side-entry sample stage. The first version of the stage has been built for Orsay's CHROMATEM project: a high energy resolution monochromated STEM-EELS (HERMES) instrument, whose requirements include cathodo-luminescence (CL) capabilities with both light detection and injection [3], and a liquid nitrogen-cooled sample holder. A CL mirror can be introduced on the entrance side of the sample, in a 6 mm OL polepiece gap. The mirror is UHV-compatible and precisely moveable, so that the mirror's focal spot can be made to coincide with the "optimum probe formation spot" of the objective lens, about 0.5 x 0.5 x 2 µm in size (w x d x h). The mirror can couple out CL, or bring in laser illumination, as needed for instance for energy-gain spectroscopy. The cathodo-luminescence capabilities of the system are complemented by Nion's sub-10 meV energy resolution ground-potential monochromator [4], and a new Nion electron energy loss spectrometer optimized for vibrational studies.

The key features of Nion's side entry stage are:

a) A sample rod with a principal external diameter of 16 mm, small enough so that changes in air pressure outside the column do not result in major sample movement. The rod width is reduced to 8 mm at the sample, but this is enough to be compatible with a wide variety of sample "services".

b) Nion's ultra-stable sample movement mechanism, which shifts the front of the sample rod via a spherical rolling joint. Motion perpendicular to the rod axis is nearly friction and hysteresis free.

c) Compact airlock and goniometer mechanism that performs just two main functions: vacuum management during sample exchange, and setting the primary (alpha) tilt within a 180° range with arcminute precision.

d) UHV design principles: all seals are either metal or have a guard vacuum; ion pumps evacuate both the high and the guard vacua; a turbo pump roughs down the airlock; minimal area of the sample rod in high vacuum; and the high-to-guard-vacuum O-rings are never exposed to air (so they don't soak up air moisture and later release it in the high vacuum). The stage can be baked at 140° C.

Figure 1 shows a schematic cross-section through the inner part of this sample stage. The front of the sample rod is moved by the stage ring in a way that is similar to Nion's cartridge stage design [1]. The motion is coupled into the rod by a spherical rolling joint. The airlock fixes the transverse position of the back of the sample holder, and instabilities affecting the airlock are reduced by 5x at the sample.

A stable liquid nitrogen holder has been developed by CrIPTA for the stage, and will be described separately. Nion plans to continue working with third parties to develop a variety of in-situ capabilities. The stage's overall performance will be reported at the meeting.

References:

[1] OL Krivanek *et al*, Ultramicroscopy **108** (2008) 179.
[2] OL Krivanek *et al*, Microsc. Microanal. **16** (Suppl 2, 2010) 70.
[3] M Kociak and LF Zagonel, Ultramicroscopy 174 (2017) 50.
[4] OL Krivanek et al., Nature **514** (2014) 209.

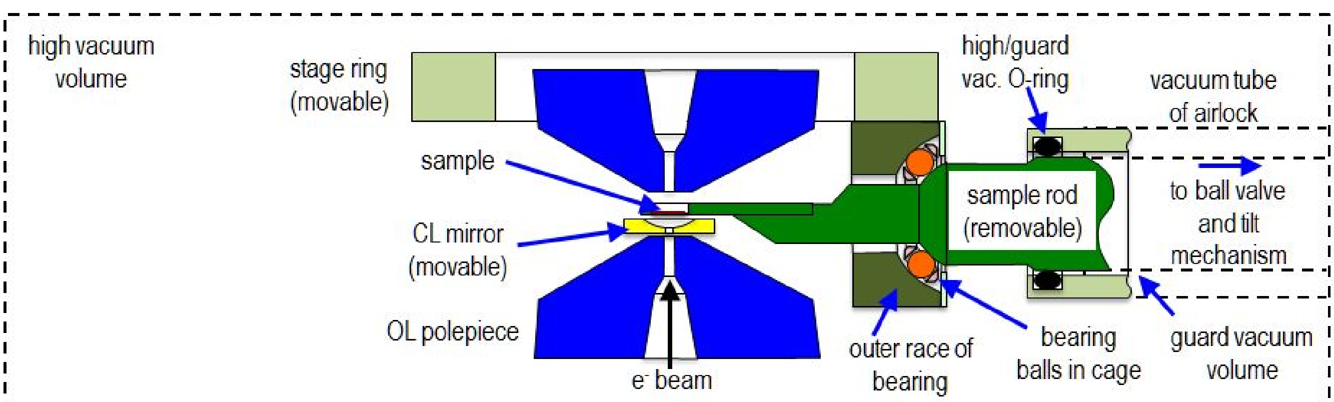

Figure 1. Schematic diagram of the Nion side-entry stage. The front part of the sample rod is moved around by the stage ring. The outer components of the stage are not shown.

https://doi.org/10.1017/S1431927617000952 Published online by Cambridge University Press